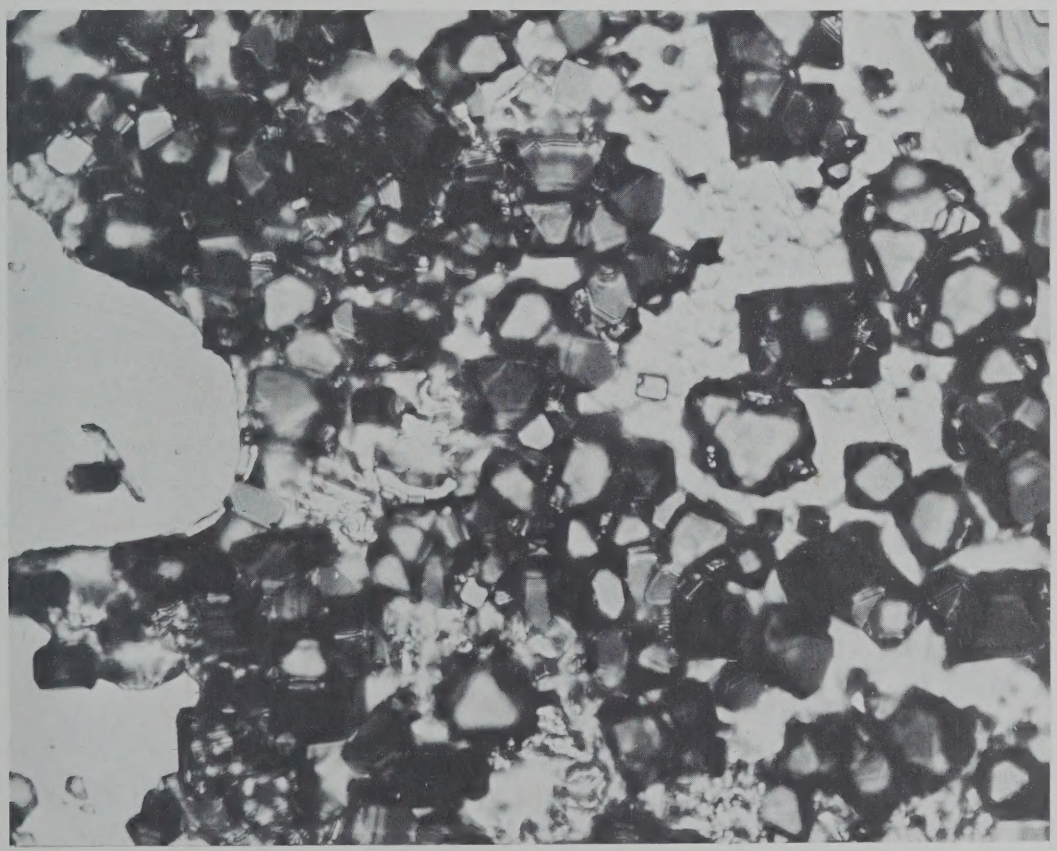
NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN



VOLUME 45 MAY 1961 NUMBER 5

U.S. DEPARTMENT OF COMMERCE — LUTHER H. HODGES, *Secretary*

NATIONAL BUREAU OF STANDARDS — A. V. ASTIN, *Director*

NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. Subscription price, domestic $1.50 a year; 75 cents additional for foreign mailing; single copy, 15 cents. Use of funds for printing this publication approved by the Director of the Bureau of the Budget (June 24, 1958).

May 1961 — Issued Monthly — Vol. 45, No. 5

Contents

	Page
Calibration of Inductive Voltage Dividers	73
Luminance Standards	75
Separation and Determination of Iron, Aluminum, Titanium, and Zirconium	76
Viscoelastometer for Measurement of Flow and Elastic Recovery	78
Polymorphism of Rare-Earth Borates	80
Low-Level Radioactivity Laboratory Established	81
Standard Materials	82
Antennas for Detecting Micropulsations	83
First Construction Bids Sought for New Laboratories	84
Point in Antarctica Named for Boulder Scientist	88
Publications of the National Bureau of Standards	89

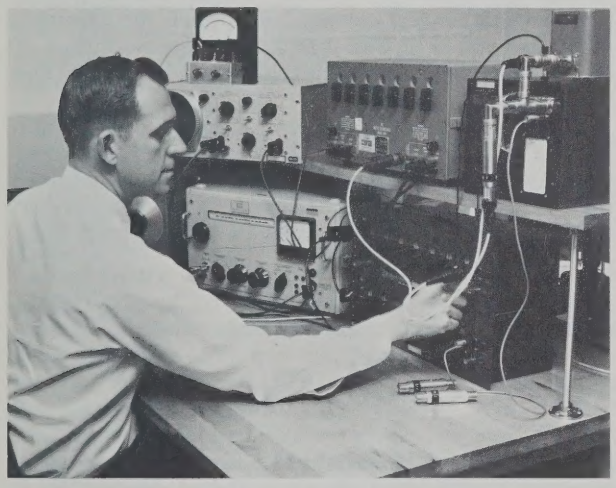
COVER: Random formation of oxide crystallites on a copper single-crystal specimen. The crystal had been exposed to pure water containing oxygen and carbon dioxide for five days. Metals in the form of single crystals are being studied at NBS to obtain information on the fundamental nature of the corrosion process. In this way, many of the corrosion-influencing factors of polycrystalline materials can be isolated and reproducible results obtained. (See "Corrosion Research," p. 150, August 1959 issue; and "Low-Temperature Reaction in Copper-Oxygen System," p. 61, April 1961 issue.) (Magnification: ×580.)

CALIBRATION OF INDUCTIVE VOLTAGE DIVIDERS

Fourth of a Series on NBS Measurement and Calibration Services

ACCURATE CALIBRATIONS of inductive voltage dividers are now available from the Bureau in Washington, D.C., and Boulder, Colo. This service, performed for standards laboratories, industrial concerns, and various research groups, plays an important part in the Bureau's basic electrical measurements program. By maintaining standards against which electrical devices can be calibrated, the Bureau insures a common, consistent basis for electrical measurements in this country.

Inductive voltage dividers in general are tapped windings on toroidal cores which have very high magnetic permeability. Decade inductive dividers are useful as ratio arms of precision bridges, as voltage dividers for the accurate division of voltages, and as standards for calibrating other dividers by a comparison method.



The Bureau has recently improved the accuracy of calibration of inductive voltage dividers. Here, R. V. Lisle of the Bureau's Boulder, Colo., laboratories adjusts the standard voltage divider to determine corrections for the divider under test.

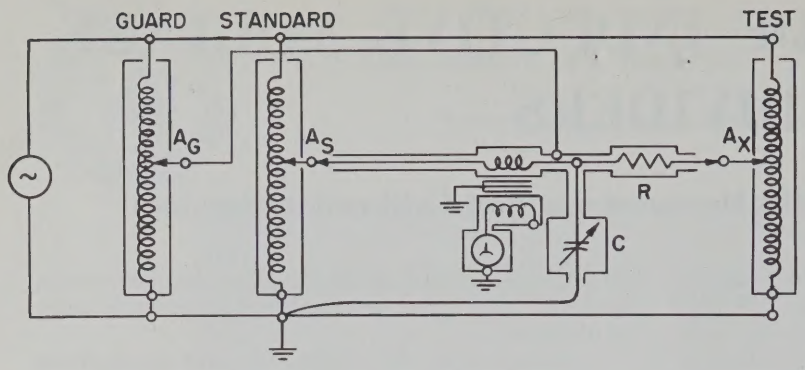
The initial establishment of voltage ratio over a wide range requires complex procedures and specially fabricated equipment. A comparison method is then used at the Bureau to provide a simple and efficient calibration service with negligible degradation of accuracy. Close attention is given to sources of error that formerly would not have been detected. The precision of this method has been verified by T. L. Zapf and R. V. Lisle of the Boulder laboratories. The method is a modification of a comparison technique investigated earlier by W. C. Sze of the Washington laboratories. The comparison method described requires very little equipment and should find widespread application in many laboratories.

A calibration that is adequate for most measurement purposes consists of a determination of the correction to the nominal output-to-input voltage ratio for each step setting of the highest three decades at a specified input voltage and frequency (commercially available inductive voltage dividers usually have four to seven decades). This procedure provides about 30 discrete ratio settings for which the true voltage ratio can be computed. For best accuracy only one of the highest decades should be set at a step other than zero at any one time because, in general, the corrections for the separate decades cannot be simply combined. However, the reading of the lowest decades usually does not significantly affect the correction to the reading of the highest decades. Thus, if the complete reading of the inductive voltage divider is defined as the sum of the reading of the highest decades plus the reading of the lowest decades, the true ratio at this setting will be this sum plus the correction to the highest decades.

A guard around the detector input circuit prevents current leakage through stray impedance which would otherwise seriously degrade the accuracy of the measurement. The guard is connected to a divider set at the same reading as the standard so that the shield is nearly at the same potential as the output of the standard and test dividers.

If the connections to ground are made as shown in the diagram, the stray capacitance from the shield to ground is predominantly from the shield surrounding the input winding of the detector transformer and the case of the variable capacitor. Voltage across this capacitance must not produce a current in the shield of the coaxial cable leading to the output terminals of the dividers, because this current would induce a voltage in the inner conductor that could produce a noticeable error; instead, the current must be conducted directly to the guard divider. Therefore, the shielded connector between the detector transformer and the capacitor is short, and the output of the guard divider is connected at this point. Care must be exercised not to touch the shield circuit because of the shock hazard, and it is advisable to insulate the coaxial cable and connectors to avoid accidental short circuits. Fuses in the output of the power supply are helpful in preventing damage from short circuits.

The possibility of error arising from the difference in voltage between the shield and ground can be avoided if the guard circuit is grounded at the output of the guard divider rather than at one end of the divider. With this connection it may be necessary to use an



Bridge circuit used in calibrating inductive voltage dividers by comparison with a standard. The input terminals of the test and standard dividers are connected with heavy, short leads to reduce voltage drops between dividers.

isolation transformer between the oscillator and the measurement circuit. The cases of the dividers will not be at ground potential and care should be taken to avoid shocks and short circuits.

The resistance and capacitance produce a component of voltage approximately in quadrature with the voltage applied to the inductive divider. This quadrature voltage can be made to oppose that resulting from the small phase angle difference between the output voltages of the standard and test inductive voltage dividers. As the resistance is much smaller than the capacitive reactance, the predominantly capacitive current produces the desired quadrature voltage across the resistance. By proper choice of resistance the phase angle difference, in microradians, can be made direct reading on the variable air capacitor marked in picofarads. It can be shown that the difference in phase angle between the outputs of the two dividers is approximately equal to ωCR, in radians.

The variable air capacitor chosen should not be large enough to load the inductive voltage dividers appreciably. The effect of this loading can be seen by placing an additional capacitor of equal value across the output terminals of the divider and noting the change in balance conditions of the bridge. An extrapolated correction for loading can easily be applied.

A phase-sensitive detector-amplifier with oscilloscope presentation can be used to good advantage. If the detector responds linearly to small voltage differences, the careful observer can interpolate between the step settings on the smallest decade. If adequate amplification is available in the detector-amplifier, the precision of comparison is limited primarily by the resolution of the standard and test inductive voltage dividers and the ability to interpolate.

A partial verification of the accuracy of the comparison method together with the accuracy of corrections applied to the standard divider can be obtained by reversing the input connections to the test divider, and measuring the complement of its indicated ratio.

Meeting on High-Precision Connectors

A ONE-DAY MEETING on high-precision connectors will be held on June 29, 1961, at the Boulder, Colo., Laboratories of the Bureau.

The objective of the meeting is to reduce those errors in precision measurements which are due to connector uncertainties in coaxial equipment at radio and microwave frequencies. It is expected that this can be achieved by agreement on design, size, tolerances, etc., of a minimum number of connectors which will be used by standardizing laboratories and incorporated in manufactured instruments capable of high precision. The morning will be devoted to papers giving quantitative information on high-precision connectors and the afternoon to a round table discussion on the same topic.

Papers and discussion are invited on such subjects as the influence of mechanical and electrical tolerances on the residual discontinuity of connectors, residual discontinuities in coaxial lines and cables, high precision coaxial lines, dielectric material for supports in connectors, support structures for connectors, power handling capacity, upper frequency limits of connectors, adapters for differing diameters and impedances, measurement methods and results, stability and wear properties of electrical metals, surface plating and maintenance, adapters to existing connectors, fabrication methods, leakage resistance, recommended sizes, characteristic impedance, frequency ranges, maximum VSWR, environmental performance, current and anticipated needs, and other topics which may aid agreement on an optimum coaxial connector for high precision work and national and international agreement.

LUMINANCE STANDARDS

IN RESPONSE to widespread requests for a precise means of evaluating the luminance or "brightness" of aircraft instrument dials and panels, the Bureau has initiated a program to provide luminance standards. Consisting of two components—a diaphragmed flashed opal glass and an electric lamp—these standards are issued with a report of calibration containing instructions for their use.[1] Because of the accuracy with which brightness measurements may be made with the aid of these standards, they are also being utilized for other standardizing purposes; for example, to achieve uniformity in the luminances of cathode ray tubes and television sets, and to calibrate the luminance meters employed by illumination engineers to measure the brightness of visual environments.

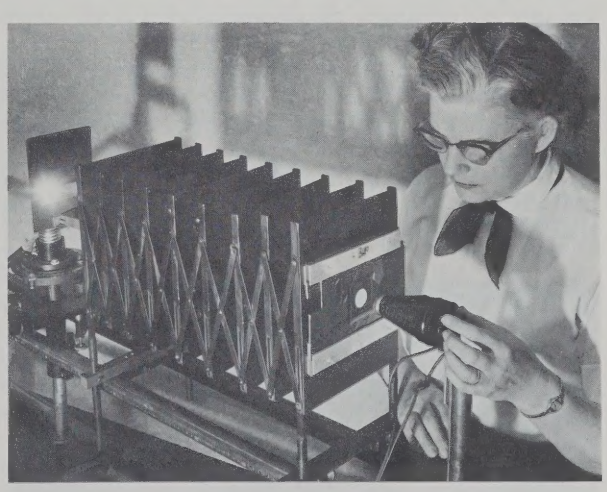
Because of the availability of flashed-opal glass, whose transmittance is reasonably high (about 50 percent) and uniform over a considerable range of angles from the normal to the glass surface, samples cut in 2-in. squares from this material were selected for calibration as one component of the luminance standards. Projection-type lamps of 100, 250, and 500 w, and inside-frosted tubular lamps[2] of 100, 300, and 500 w, are calibrated for luminous intensity as the second component. Before calibration, both the glasses and the lamps are tested for their suitability for issuance as these components.

In the calibration report, the value for the luminous intensity of the lamp component is given as the candlepower of the lamp in a specified direction, and the value for the luminous directional transmittance of the glass component is given as the number of footlamberts emitted by the diaphragmed side of the glass per footcandle incident on the undiaphragmed side. The incident footcandle value is calculated from the luminous intensity of the lamp and the distance between the filament of the lamp and the glass. The two components are readily adapted to calibrating any type of photometer. The photometer may then be used directly to measure the luminance of achromatic light-diffusing surfaces, and with a color-matching filter of known transmittance as an additional component, to measure the luminance of chromatic surfaces.

The calibration of the lamp component at the Bureau is accomplished by means of an instrument that utilizes a thermopile as a photometric receiver.[3] In this procedure, the lamp is oriented on a photometric bar at a prescribed distance from the receiver. Each projection-type lamp is appropriately diaphragmed so that light from only its filament is transmitted to the receiver. When a specified voltage is applied to the lamp, values for determining the luminous intensity of the lamp are automatically recorded. The factor used for determining luminous intensity from the recorded values is determined at regular intervals by calibrating the receiver with a group of lamps that are the national reference candlepower standards.

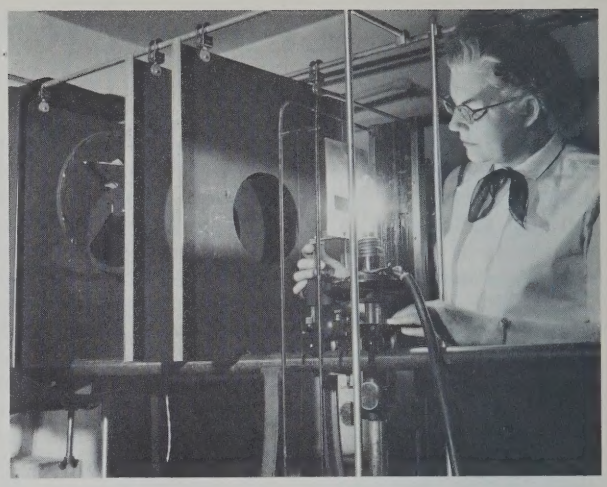
When the glass samples are prepared for calibration, the flashed side of each glass is taped to a diaphragm with a centrally located circular opening 1 in. in diameter. A lamp operated at a constant voltage and the glass sample are inserted in holders and positioned on a photometric bar at least 1.5 ft apart; an array of baffles is arranged between the lamp and glass to eliminate stray light. A device containing a photoelectric cell[4] and a ¾-in.-diam diaphragm is then placed close to the diaphragmed (flashed) side of the glass, and the photoelectric current, which is proportional to the luminous directional transmittance of the glass, is recorded. The equipment used for this procedure is calibrated against a master set of standards, consisting of three squares of flashed opal glass similar to those issued as laboratory standards. The luminous directional transmittance of the master standards was determined as follows: The illumination in footcandles incident on the undiaphragmed side of each glass was set at a known value and measurements were made of the luminous intensity in candles of the diaphragmed side; the luminance in footlamberts of this side was then computed from the measured luminous intensity divided by the area in square feet; finally, the luminous directional transmittance of the glass was computed as the ratio of this luminance in footlamberts to the footcandles incident on the undiaphragmed side.

The flashed opal glass, a more stable component than the lamp, needs recalibration at very infrequent intervals. The lamp, however, should be recalibrated after each 10 hr of use until it is found that longer periods between recalibrations are adequate. One measure of



Mrs. Velma I. Burns adjusts a device containing the photoelectric cell used in calibrating the flashed-opal glass component of luminance standards. Light from the filament of a lamp (left) is diffused through the circular opening of the diaphragmed side of the glass onto the photocell.

Photometric equipment used to calibrate the lamp component of luminance standards. Mrs. Burns adjusts a movable diaphragm so that light from only the filament of a projection-type lamp is transmitted to the receiver (behind shield at extreme left).



lamp deterioration is a change from the reported value in the amount of current consumed by the lamp when operated at a specified voltage.

[1] *Luminance standards may be procured at prices ranging from $63.00 to $72.00 (depending on the size of the lamp component) from the Photometry and Colorimetry Section, National Bureau of Standards, Washington 25, D.C. Information on other NBS calibration services may be obtained from the NBS Test Fee Schedule, available upon request from the Publications Section, NBS.*

[2] Vertical distribution of light from gas-filled candlepower standards, *by L. E. Barbrow and S. W. Wilson, Illum. Engr.* **53,** *645 (1958); and New candlepower* standards, *NBS Tech. News Bul.* **43,** *163 (1959).*

[3] A physical photometer, *by R. P. Teele, J. Research NBS* **27,** *217 (1941) RP1415.*

[4] A photometric procedure using barrier-layer photocells, *by Louis E. Barbrow, J. Research NBS* **25,** *703 (1940) RP1348.*

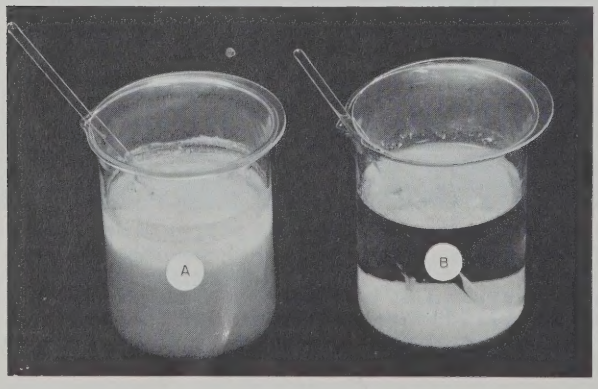
Separation and Determination of Iron, Aluminum, Titanium, and Zirconium

TITANIUM, zirconium, iron, and aluminum can now be effectively separated and quantitatively determined by an analytical method [1] developed by the Bureau for the Diamond Ordnance Fuze Laboratories. By starting with a ½-g sample of mixed oxides and by following this procedure, workers with proper technical training can determine the amount of each of the metals present within ±0.2 mg.

Separation of these four elements has been a difficult problem for many years. However, it was found that by combining conventional and improved techniques, sharp separations of these metals can be obtained.

The method was developed by T. J. Murphy, W. S. Clabaugh, and R. Gilchrist of the inorganic chemistry staff as part of a continuing program to develop analytical techniques and to produce purer reagent chemicals.[2] Although originally designed for the quantitative analysis of barium titanate dielectrics, it is applicable to the analysis of alloys and other materials containing the four metals.

In general, the procedure consists of five main steps. First, the aluminum, iron, titanium, and zirconium are collectively precipitated with 8-hydroxyquinoline. Second, the precipitated quinolates are dissolved in a solution containing citric acid and hydrochloric acid, from which the iron is precipitated with 1-nitroso-2-naphthol. Third, the titanium and zirconium are collectively precipitated with cupferron. Since only aluminum remains in the filtrate, it can be precipitated with cupferron at *p*H 4.0. Fourth, the titanium and zirconium cupferrates are decomposed, and then redissolved in sulfuric acid. Next, the zirconium is complexed with ethylenediaminetetraacetic acid and the titanium precipitated with 8-hydroxyquinoline at a final *p*H of 4.5. Finally the zirconium is reprecipitated with cupferron.



As one part of the process, aluminum is separated from titanium and zirconium by cupferron reagent. Ordinarily, a bulky flocculent precipitate results (left) and aluminum coprecipitates. However, in the Bureau method, slightly less than the stoichiometric amount of cupferron solution is initially added, and the solution is heated to 60 °C. Then the precipitated cupferrates change to a dense, compact form (right), making a sharp separation possible.

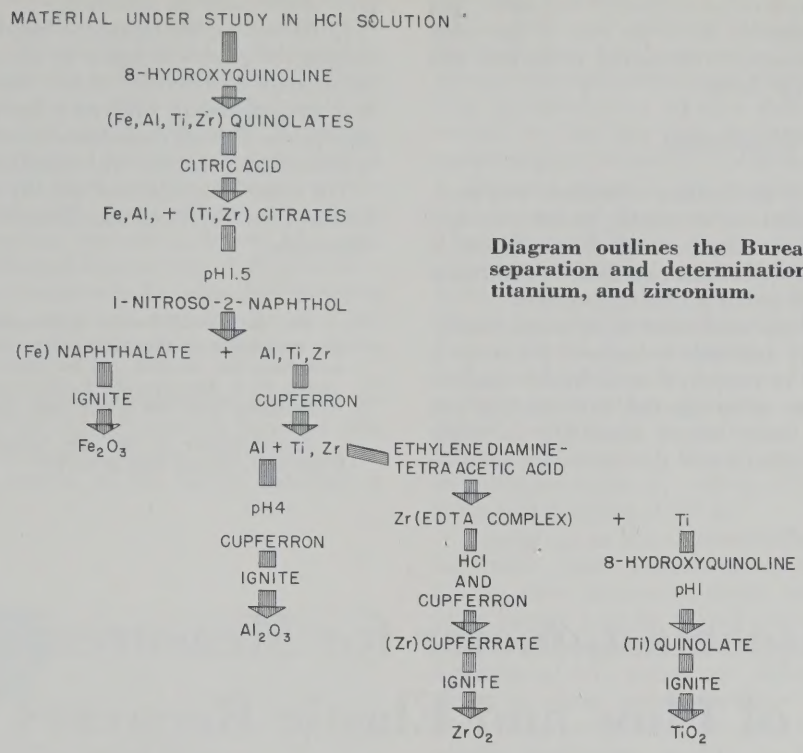


Diagram outlines the Bureau's new method for separation and determination of iron, aluminum, titanium, and zirconium.

Procedure

To begin the separation, an unknown specimen is dissolved by digestion in hydrochloric acid. The four elements—iron, aluminum, titanium, and zirconium—can then be collectively isolated by precipitation with 8-hydroxyquinoline. The precipitation sharply separates the four elements from the alkalies, alkaline earths, and other undesirable substances. The resulting quinolate precipitates are dissolved in a solution of citric acid and dilute hydrochloric acid.

When an alcohol solution of 1-nitroso-2-naphthol is added to the solution containing the four elements, the ferric ion is quantitatively precipitated. It can then be ignited to ferric oxide and weighed. Aluminum does not precipitate with 1-nitroso-2-naphthol. The citrate ion forms complexes with zirconium and titanium and prevents these two metals from precipitating.

To separate aluminum from zirconium and titanium, first hydrochloric acid and then cupferron reagent are slowly mixed in the filtrate obtained from the iron removal. Less than the stoichiometric amount of cupferron—only enough to precipitate 90 percent of the ziconium and titanium—is added. Then the solution is heated to about 60 °C. This heating causes the precipitated cupferrates to change from a bulky, flocculent form to a dense, compact mass. After the cupferron solution has been thoroughly heated, the precipitation is completed by slowly adding more cupferron until a permanent precipitate no longer forms. Finally, cooling the solution and mixing in a slight excess of cupferron results in a clean separation. The heat treatment and the careful addition of cupferron reduces the aluminum co-precipitation.

The aluminum present in the original specimen is determined by adding dilute ammonium hydroxide to the filtrate obtained from the preceding cupferron precipitation until a *p*H of 4.0 is reached. The aluminum is quantitatively precipitated by cupferron from this weakly acid solution. After ignition in an electric furnace, the residue is weighed as aluminum oxide.

In the last part of the analysis, ziconium and titanium are separated from each other. After the zirconium and titanium cupferrates have been separated from the aluminum solution, they are decomposed at 450 °C to the oxides. Next, to dissolve these titanium and zirconium oxides, dilute sulfuric acid is added. Following this step, an excess of ethylenediaminetetraacetic acid is added to form a strong complex with zirconium and a weaker one with titanium. Then, an excess of solid 8-hydroxyquinoline is mixed in and the *p*H of the solution carefully and gradually adjusted to 4.5. Although the zirconium complex is strong enough to prevent the precipitation of zirconium by the quinoline reagent, titanium is completely precipitated as titanyl quinolate.

For the subsequent titanium determination, the titanyl quinolate is decomposed and then ignited in an electric furnace. The resulting residue is weighed as titanium dioxide.

Zirconium can be quantitatively precipitated by cupferron from a hydrochloric acid solution in the presence of ethylenediaminetetraacetic acid. Thus, following the titanium removal, hydrochloric acid is introduced

into the remaining solution. Then, slightly more than the stoichiometric amount of cupferron is added, and the zirconium precipitates. As in the case of the other three metals, the zirconium compound is ignited and determined in its oxide form.

Evaluation

The effectiveness of the Bureau procedure was evaluated by analyzing solutions containing known amounts of the four metals. From this controlled experiment, it was found that the procedure can be used to determine a total quantity of about 0.5 g of mixed oxides.

The reagent chemicals used must be of good quality. Because commercially available samples of 1-nitroso-2-naphthol were found to contain a considerable amount of residual impurities when ignited, this reagent was leached with warm water before dissolving in dilute ethanol. Both this solution and the cupferron solution should be freshly prepared just before analysis because they decompose on standing.

Ethylenediaminetetraacetic acid is purified by dissolving the practical grade of the acid in a solution of ammonium hydroxide. After this solution is filtered, it is neutralized to *p*H1 with hydrochloric acid to reprecipitate the ethylenediaminetetraacetic acid, which is filtered and washed with distilled water.

The other reagents used should conform to the specifications [2] of the American Chemical Society for reagent chemicals.

---

[1] *For further technical information, see* Method for the separation of titanium, zirconium, iron, and aluminum from one another and for their subsequent determination, *T. J. Murphy, W. S. Clabaugh, and R. Gilchrist, J. Research NBS* **64** *(Phys. and Chem.) 535 (1960).*

[2] *Specifications of the Am. Chem. Soc. for Reagent Chemicals, revised edition (1961).*

Viscoelastometer for Measurement of Flow and Elastic Recovery

A RECORDING VISCOELASTOMETER has been developed by the Bureau under the sponsorship of the Office of Naval Research and the Office of Synthetic Rubber of the Federal Facilities Corporation.[1] This instrument, designed by Richard J. Overberg and H. Leaderman, consists essentially of a concentric-cylinder rotational viscometer, having a fixed outer cylinder and a rotatable inner cylinder. A constant torque is applied to the rotor, and subsequently released, in a stepwise manner. It has been used to measure the linear viscoelastic behavior of such polymers as polyisobutylene and polystyrene, and can also be used to determine the viscoelastic properties of asphalt, roofing tar, and, with modification, vulcanized rubber. Viscosity measurements can be made in the range of 10^6 and 10^{12} poises, and the elastic recovery determinations recorded from 1 sec to 1 day. Measurements can be made on samples exhibiting non-Newtonian flow, provided the apparent viscosity is not much less than a Newtonian viscosity.

A direct method for measuring the viscoelastic properties of natural and synthetic rubbers has been needed in order to make optimum use of these materials. Although it is possible to calculate the viscosity and elastic recovery of polymers from the data obtained in other experiments, the results will not be reliable unless the initial data are extremely accurate. The viscoelastometer was therefore developed to provide, directly, a means of investigating these viscoelastic properties.

When a constant torque is applied to the rotor, there occurs a rotation, the angular velocity of which gradually decreases to a constant value. This rotation is due to a viscous flow upon which is superimposed a retarded elastic deformation. Upon removal of the torque, there occurs a gradual elastic recovery. The terminal portion of the retarded elastic component is dominated by the flow, hence it cannot be measured directly; it is obtained from the retarded elastic recovery by procedures developed from the time-temperature equivalence principle and the theory of linear viscoelasticity.[2]

These procedures were applied to data on several samples of polyisobutylene of low molecular weight in an earlier instrument.[3] For the practical range of torques in the viscoelastometer, the behavior may range from slightly to severely nonlinear. For example, if the torque is doubled, the flow rate is more than doubled, while the terminal portion of the elastic recovery curve is increased by a factor less than two, and linear behavior is obtained by extrapolation. When the rheological behavior is significantly nonlinear, the liquid tends to climb up the rotor due to secondary flow. To guard against this, the stator is a precision bore glass cylinder so that the free surface of the liquid may be continuously observed.

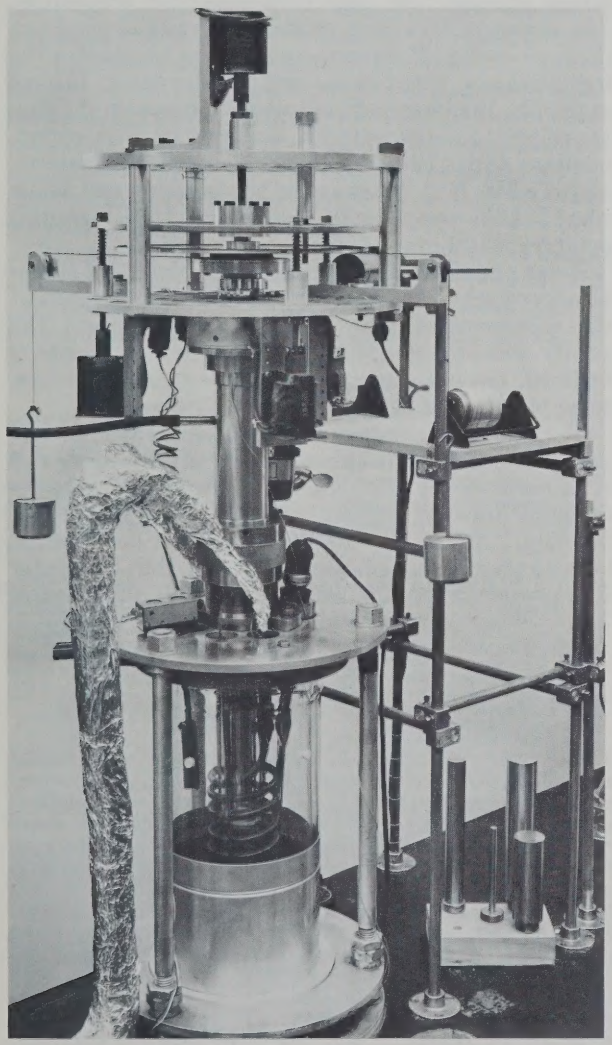
The determination of viscosity with this instrument depends upon the constant rate of rotation of the rotor subsequent to the dissolution of the elastic component of the material. The steady state elastic compliance is then determined from the magnitude and rate at which the rotor "unwinds" after the torque is removed. The material being tested is placed in a precision bore cylindrical glass stator, and a concentric rotor, attached to

a shaft, is partially immersed in the sample. As any eccentricity of the rotor's motion would result in inaccurate results, the shaft is accurately centered with precision bearings. Torque is applied to the shaft by means of weights suspended from lateral pulleys. These weights are so arranged as to apply a pure torque to the shaft. The angular velocity of the shaft is proportional to the applied torque minus a constant friction torque. The amount of shaft rotation, during both the application of torque and elastic recovery, is determined by a photoelectric recording device. An incident light beam is reflected from a ring of 90 mirrors—each subtending 4°—fastened to the outside of the rotor shaft. The reflected rays are detected by a light-sensitive cell, which controls the pen position on a strip chart recorder.

The application of torque initiates the viscosity determination. When the rate of rotation becomes constant with time, as indicated by the chart, the torque is removed, and a record of the elastic recovery is produced on the same chart. The viscosity determination may require from 1 sec to 24 hr, and elastic recovery will cover a similar period. When the recovery determination time is excessive, the chart drive mechanism is operated intermittently by a preset timing device. The reproducibility of this determination is demonstrated by the fact that duplicate runs on the same material agree to within ± 2 percent.

The temperature of the sample is controlled by placing a constant temperature bath around the stator. Thermal equilibrium is checked by thermocouples placed inside the bath and inside the rotor. The leads from the rotor's thermocouple terminate in mercury slip rings, to reduce static friction to a minimum. Heat flow to the shaft is minimized by an insulating glass link placed between the rotor and the shaft. However, a slight temperature gradient does exist through the liquid, which must be corrected for during final calculations of results. Runs have been made over the temperature range of -40 to $+160$ °C, with temperatures held to within 0.02 °C.

Depending on the characteristics of the material being tested, such parameters as rotor diameter, rotor length, rotor immersion depth, end clearance, and applied torque may be varied considerably. The sensitivity of the apparatus is changed through the use of interchangeable rotors of different diameter. The range in shear stress available is 10^3 to 10^6 dyne/cm^2. The practicable range for measurement of elastic compliance is 10^{-4} to 10^{-7} cm^2/dyne. The end clearance—the distance between the end of the rotor and the bottom of the stator—is varied by means of gage blocks used to support the shaft. The effect of end clearance was found to be inversely proportional to the amount of clearance. This relationship conforms to theoretical expectations, but the rate of change with varying end distance is much greater than predicted.



Viscoelastometer developed by the Bureau for determining the viscosity and elasticity of liquid polymers. Sample is placed in the glass stator (inside coil, center bottom) and a rotor, attached to the shaft, is partially immersed in the sample. A known torque is applied to the shaft by means of lateral pulleys. The rate at which the torque turns the rotor, and then the rate at which the rotor "unwinds" after removal of the torque, are automatically registered by a photorecorder. Light is reflected from a ring of mirrors, each subtending four degrees (near top of shaft), to twin photocells on the pen carriage of the strip-chart recorder.

---

[1] *For further details, see* Viscoelastometer for measurement of flow and elastic recovery, *by Richard J. Overberg and H. Leaderman, J. Research NBS* **65C** *(Eng. and Inst.) 9–21 (1961).*

[2] Rheology of polyisobutylene I, *by H. Leaderman, J. Polymer Sci.* **13,** *371 (1954).*

[3] Rheology of polyisobutylene II, *by H. Leaderman, R. G. Smith, and R. W. Jones, J. Polymer Sci.* **14,** *47, (1954).*

591904 O - 60 - 2

POLYMORPHISM OF RARE-EARTH BORATES

THE POLYMORPHIC STATES of rare-earth borates have been studied by the Bureau. This investigation,[1] conducted by E. Levin, R. Roth, and J. Martin, has shown that the crystalline forms of these compounds closely resemble the three polymorphs of calcium carbonate—calcite, aragonite, and vaterite. No vaterite structure, other than calcium carbonate, had previously been known. X-ray diffraction studies of the borate-type vaterites has led to the conclusion that the poorly defined vaterite form of calcium carbonate is probably pseudo-hexagonal.

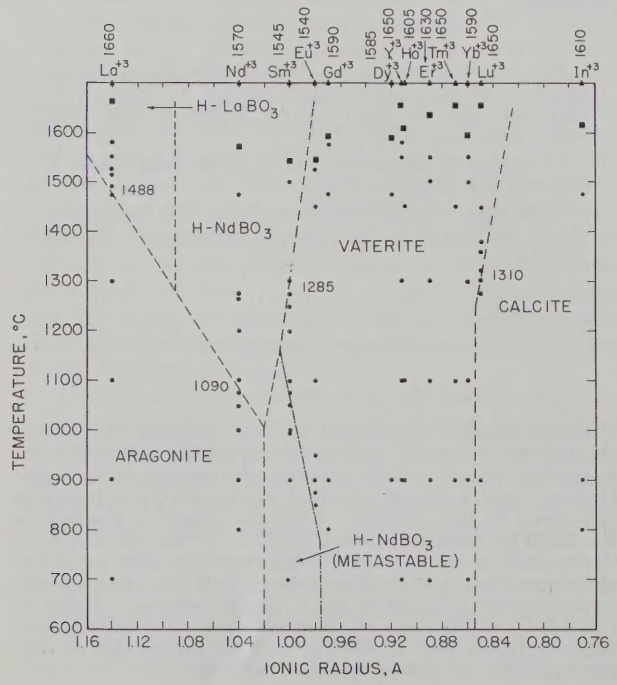
The increasing availability of highly-purified rare-earth oxides has stimulated the investigation of systems containing these compounds. The present program is part of a study of the binary phase equilibrium relationships existing between boric acid and trivalent rare-earth oxides. The lanthanide (rare-earth) series is significant in that it consists of a large group in which the ionic radii form a continuous series from lanthanum (1.15A) to lutetium (0.85 A). Because their crystal structures are similar to those of the rare earth oxides being studied, the oxides of lanthanum, yttrium, manganese, indium, and scandium were also included.

Compounds equimolar in rare-earth oxide and boric oxide, having the general formula ABO_3, were prepared for a study of their crystalline forms. Calculated amounts of boric acid and trivalent rare-earth oxide were mixed in a high-speed mechanical blender for 30 min, pressed into disks at 20,000 psi, and fired in platinum crucibles. The disks were ground, pressed, and fired twice more prior to further study. These compounds were then held at specified temperatures until equilibrium was achieved, and then quenched. X-ray, infrared, and polarizing microscope examinations were then made to determine which polymorph was present.

The most striking result of this study is the finding that the rare-earth borates possess the same crystal structure types as the three polymorphs of calcium carbonate. However, none of the borate compounds were found in all three forms. Lutetium borate was produced in the calcite and vaterite form, whereas the others showed only one carbonate-type polymorph. The structure type of a particular borate depends on the ionic radius of the rare-earth cation involved. The large cations (lanthanum and neodymium) formed aragonite, the intermediate cations (europium through ytterbium) formed vaterite, and the smallest cation, lutetium, forms calcite at low temperature and vaterite above 1,310 °C. Lanthanum, neodymium, and samarium borates also formed new, unidentified high-temperature structures.

X-ray examination showed that the calcite and aragonite-type borates conform to the well-known patterns of the analogous calcium carbonate polymorphs. The poorly defined vaterite phase of calcium carbonate is believed, through comparison with rare-earth vaterites, to be pseudo-hexagonal.

The infrared patterns of rare-earth aragonite and calcite forms are analogous to the corresponding calcium carbonate forms.[2] However, the vaterite patterns are quite different, indicating differences in the oxygen bonding. Apparently the BO_3 groups in the vaterite-type borates are not planar, as contrasted to the planar CO_3 groups in calcium carbonate. This difference in



In a study of rare-earth borates having the general formula ABO_3, the Bureau found that polymorphs corresponding to the three forms of calcium carbonate—calcite, aragonite, and vaterite—are formed by these compounds. In this diagram, the ionic radii of the rare earths are plotted versus temperature. Melting points are listed next to the symbols at the top. Dashed lines indicate boundaries between adjacent ABO_3 phases. Dash-dot line indicates the limit of formation of the metastable high-temperature form in the vaterite field. Lutetium (Lu) was the only borate found in two of the calcium carbonate forms—calcite below 1,310 °C and vaterite above this temperature. From this study the structure of vaterite is believed to be pseudo-hexagonal.

orientation may account for the fact that the borate vaterites are stable, whereas the carbonate vaterite is metastable.

One potential application of this research is in the field of high-temperature ceramics. The melting points of the rare-earth borates are above that of boric oxide, and phase studies have shown that some of these compounds exist in equilibrium with liquid boric oxide above 1,400 °C. Thus a rare-earth borate ceramic could be used where resistance to attack by boric oxide is important.

[1] *For further details, see* Polymorphism of ABO_3 type rare-earth borates, *By E. Levin, R. Roth, and J. Martin, Am. Minerologist* (to be published).

[2] Infra-red studies of aragonite, calcite, and vaterite type structures in the borates, carbonates, and nitrates, *by C. E. Weir and E. R. Lippincott. J. Research NBS,* **65A** (Phys. and Chem.) (1961).

Low-Level Radioactivity Laboratory Established

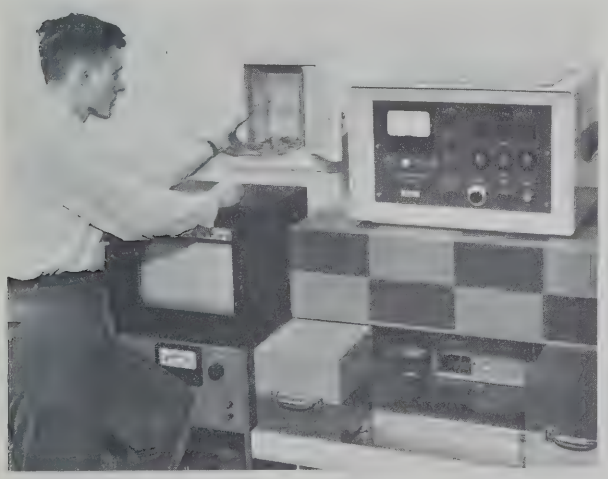
THE BUREAU has recently completed a new laboratory for the measurement of very low levels of radioactivity—down to 10^{-12} curie. A group headed by W. F. Marlow and L. L. Stockman will use these facilities to prepare accurate radionuclide standards for a number of scientific and industrial applications.

In the past several years, measurements of very low levels of radioactivity have become more numerous and more exacting in such fields as archeological dating, biological and medical studies, and health physics. Because of the increased activity and interest in these fields, a thorough investigation into the radioactive contamination of reagent chemicals and materials used in radiation detection has become necessary, and a demand for radioactivity standards at very low concentrations has arisen.

To meet this situation, an existing room in the Radioactivity Section has been extensively renovated and modified to furnish a low-level radiochemistry laboratory and a source preparation room. An airlock has been built at the entrance to this room, and air-filtration and air-conditioning arrangements are used to provide a clean, dust-free atmosphere. Although the equipment was designed to remove 99.9 percent of all dust particles larger than 1 μ in diameter, preliminary tests by Carl W. Coblentz have found no particles larger

Above: "White" room constructed for use as a low-level radioactivity laboratory and a sample preparation room. A double-door airlock and air filtration and purification are used to provide a dust-free atmosphere. Although the equipment was designed to remove 99.9 percent of all dust particles greater than 1 μ in diameter, preliminary tests have found no particles greater than 0.1 μ in diameter. Patricia Mullen (right) and Mary Lynch are preparing samples having activities as low as 10^{-10} to 10^{-12} curie/g.

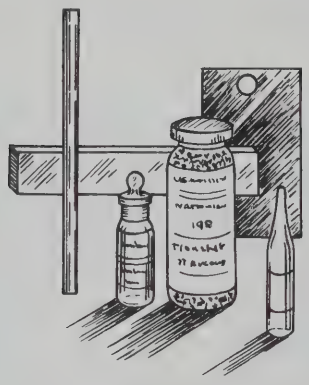
Below: Low background beta counter is used to determine the activity of specially prepared samples. E. L. Hendricks removes samples from an airlock which connects the sample preparation room with the counting room.



than 0.1 μ in diameter. Special clothing and shoes or plastic bootees are worn in these rooms.

Work is in progress on methods of measuring the amounts of radionuclides present at very low concentration (10^{-10} to 10^{-12} curie/g) and on preparing standards of radioactivity at these concentrations. Radionuclides under present investigation are natural uranium, radium 226, thorium 230, strontium-yttrium 90; other nuclides to be investigated at the earliest opportunity include sulfur 35, cobalt 60, ruthenium 106, and cesium-barium 137. The general program includes the determination of low levels of radio-contamination of materials and reagent chemicals by any nuclide, and, possibly, the preparation of materials and reagent chemicals free from any detectable radioactivity.

The counting room is adjacent to the source preparation room and is connected by a small wall air-lock through which samples may be passed. This room is equipped with low-level alpha, beta, and gamma detectors shielded either by mercury, iron, lead, or anticoincident counting circuits or a combination of two or more of these. A gamma-ray spectrometer consisting of a sodium iodide (thallium-activated) well crystal and a 100-channel pulse-height analyzer is available for the identification of radiochemical impurities in chemical reagents.



STANDARD MATERIALS

Surface Flammability Standard

AS PART OF A PROGRAM to provide improved methods for measuring the fire-hazard properties of materials, a standard material of prescribed flammability has been made available by the Bureau. This surface flammability standard, having a flame spread index of 150, aids in providing an overall check of the installation procedures used in establishing the radiant panel test apparatus.[1] In addition, by comparing the results of the three given properties of the standard, any deviation in the detailed test procedures can be evaluated. However, the standard is not recommended as a substitute for these calibration procedures.

The standard material is a representative sample from a uniform lot of tempered, fibrous, felted hardboard with one smooth side. A certificate listing three properties of this sample—flame spread index, heat evolution factor, and smoke deposit—are included with the standard material. These properties are measured by performing the test on the smooth side of the material. Prior to calibration, the material must be dried for 24 hr at 160 °F and then conditioned to equilibrium at 73 $\pm$5 °F and 50 $\pm$5 percent relative humidity.

The radiant panel method[2] of measuring surface flammability of materials, previously developed at the Bureau, has been adopted by the American Society for Testing Materials for research and development work as a tentative standard method of test, E–162. It is a comparative method applicable to building materials—especially those used as internal finishes—over which flames of accidental fires could travel. These include materials in the form of boards, sheets, and certain heavy fabrics; and finish materials applied as liquids, films, or sheets, or combinations of these with or without intermediate adhesive substances.

The essential components of the test equipment are a radiant panel with its associated gas and air supply, a specimen holder and pilot burner, a stack and thermocouple assembly, a smoke sampling assembly, an exhaust hood, a radiation pyrometer, and associated recording equipment. The radiant panel, a section of porous refractory, is mounted in a vertical plane and radiates energy at the same rate as a blackbody at 670 °C (1,238 °F). This panel provides a means for simulating the heat feedback from flames to the unignited portions of the specimen under test.

For checking the operational procedures, the standard material, backed with a sheet of asbestos board, is placed on a supporting frame inclined 30° to the vertical panel surface. Thus, the intensity of radiation at the specimen's surface is practically uniform across its width, but varies along its length. A pilot flame of acetylene premixed with air, ignites the specimen at the top. The progress of the flame front is followed by observing the times at which flames arrive at marked 3-in. intervals along the specimen length. The temperature rise of the stack thermocouples is a measure of the rate of heat evolution by the specimen.

These measurements are combined in an empirical relationship to provide a flame-spread index for the specimen. An index resulting from test of the surface flammability standard checking the certified value within specified limits indicates appropriate apparatus installation and calibration procedures. Although the amount of smoke evolved is not a factor in determining flame spread index, it indicates the extent of a parallel hazard.

A set of four specimens of surface flammability standard 1002 and accompanying certificate with experimental data may be purchased for $8.00 from the Standard Sample Clerk, National Bureau of Standards, Washington 25, D.C.

Standard Materials Reissued

Several standard materials which have been out of stock have been renewed. A low-carbon steel (166b) and a copper-nickel-chromium cast iron (115a) may now be obtained for $4.50 and $6.00 respectively per 150-g sample. In addition, the depleted stock of styrene-butadiene standard sample (386b) has been reissued. This renewed standard is now reduced in price to $32.00 for a 34,000-g sample. All three standard materials may be obtained through the Standard Sample Clerk.

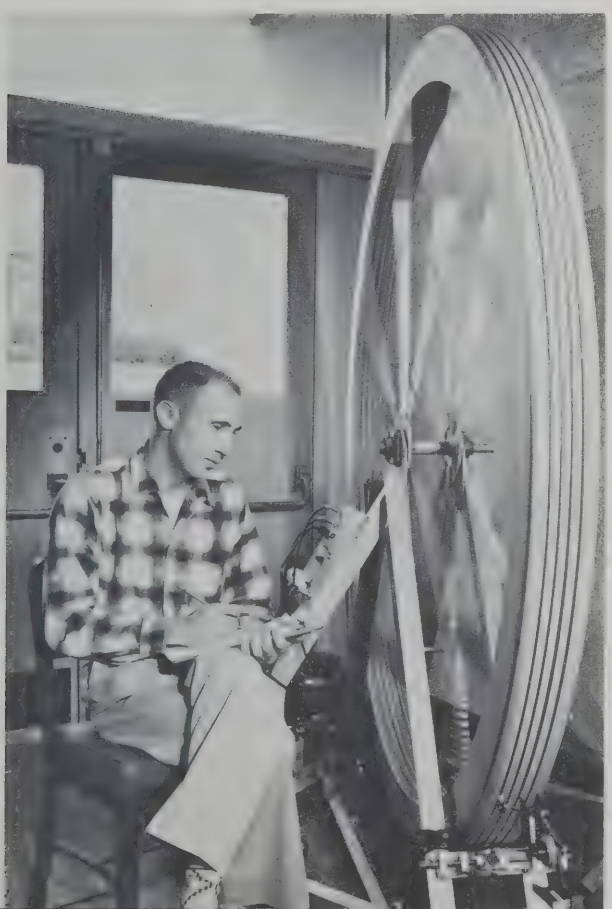
---

[1] *Interim Federal Standard No. 00136, July 31, 1959.*
[2] *For further information of the radiant panel test, see* Radiant panel test for flame spread, *NBS Tech. News Bul.* **39,** *142 (1955).*

Antennas for Detecting Micropulsations

SPECIAL-PURPOSE ANTENNAS for investigating micropulsations in the earth's magnetic field have been designed and constructed by the Bureau. These loop antennas, each 6½ ft in diameter and containing 32,000 turns of nylon-coated copper wire, will be used by W. H. Campbell of the Bureau's Central Radio Propagation Laboratory, Boulder, Colo., to collect data on the behavior of micropulsations and to discover how they can be related to unusual manifestations of solar or magnetic activity. Results of this program, only recently begun, are expected to provide further useful information about ionospheric disturbances arising from extraterrestrial sources which may affect radio communications.

Oscillations of the earth's magnetic field may be divided into three general period groups: (1) The giant pulsations, often observed on standard magnetometer records, which generally have periods of about 1 to 3 min and amplitudes reaching 10 or so gamma; (2) micropulsations with oscillation periods ranging from 5 to 30 sec and with amplitudes reaching several gamma; and (3) oscillation periods in the range 0.5 to 3 sec, which appear more rarely than the other two groups, with amplitudes occasionally reaching 1 gamma. The outstanding features of the three groups are their nearly sinusoidal form and the fact that the group characteristic periods and average amplitudes both decrease roughly by powers of ten.



Donald Wasmundt supervises the winding of one of the nine 6½-ft-diameter loop antennas which the Bureau will use to study micropulsations in the earth's magnetic field. These pulsations, of very small amplitude, can have periods ranging from 0.1 to 200 sec.

On the rim of each loop antenna, in four separate channels, is wound 130 miles of copper wire. The copper wire is covered by a layer of copper screening, and finally the whole antenna is coated with fiber glass for protection and waterproofing. This protection is necessary because the antennas will be buried in the earth to prevent interference from wind action. Sensitive enough to respond to faint, ultra low frequency electromagnetic waves or micropulsations, the antennas will be used to study oscillations in the broad range of periods from 200 sec to 0.1 sec which can be observed on a world-wide basis.

The signals now under most intensive study by the Bureau are the oscillations of from 5- to 30-sec periods. These micropulsations have been known to the scientific world since 1861, but little is understood about their origin or why the oscillations occur as they do. It is known that the signals are much stronger in the auroral zones and that they are probably associated with the influx of primary electrons into the ionosphere after the occurrence of solar storms.

Permanent micropulsation installations, using one antenna each, are being established near Boulder, Colo.; College, Alaska; and Huancayo, Peru. Six additional antennas will be used in the future to equip two portable stations where research will be conducted to measure and define the direction of micropulsations over broad areas.

Although the antennas will be used primarily in the study of micropulsations, they will also be of value in the study of two other types of electromagnetic waves—extra low-frequency sferics, which originate from lightning discharges, and 1- to 3-min giant pulsations which are thought to be caused by the resonance of the outer atmosphere when bombarded by charged particles from the sun.

FIRST CONSTRUCTION BIDS SOUGHT FOR NEW NBS LABORATORIES

ANNOUNCEMENT MADE ON BUREAU'S 60th ANNIVERSARY

Architect's drawing of the boiler plant to be constructed at the Bureau's Gaithersburg, Maryland, site.

SECRETARY OF COMMERCE Luther H. Hodges announced on March 3 that bids were being accepted for the construction of the first two buildings of the Bureau's new laboratories to be built near Gaithersburg, Md.

The Secretary said, "In line with the Administration's policy of accelerating construction activities, I am pleased that this important Federal project is ready to receive estimates from prospective builders. It is also of interest to note that this significant event in the development of improved facilities for the National Bureau of Standards occurs exactly 60 years after it was established on March 3, 1901."

Construction of the two buildings, the Engineering Mechanics Laboratory and the boiler plant, will be the first major step in the initial phase of the total relocation of the Bureau from its present site in northwest Washington. The GSA Public Building Service, which is responsible for the design and construction program, will receive the bids, and will award a contract during the month of May.

A total of $23.5 million was appropriated by Congress this year for the initial phase, which also includes site preparation, construction of a radiation physics laboratory, and the equipping of the laboratories.

The research equipment for the Engineering Mechanics Laboratory will be obtained under separate contracts. Facilities in this laboratory will include a gravity chamber; dead weight testing machines of 100,000-, 300,000-, and 1,000,000-lb capacities; a vertical tension and compression testing machine capable of loads up to 10,000,000 lb. and an Emery horizontal testing machine capable of 2,300,000-lb loads.

The total relocation program includes the construction and equipping of modern research laboratories to replace those now located in Washington, D.C., plus additional facilities for vital new programs within the Bureau's area of responsibilities. Among these new facilities being planned are a linear accelerator and a nuclear research reactor. The Bureau has estimated the total cost of the entire building program over the next several years at $104 million.[1]

Secretary Hodges visited the Bureau during the afternoon of March 3 to deliver his personal congratulations at the Bureau's anniversary celebration. Earlier, Assistant Secretary of Commerce Hickman Price, Jr., delivered the Secretary's greetings to a group of distinguished visitors at the anniversary luncheon presided over by Dr. A. V. Astin, NBS Director. Guests at the celebration included senior officials of the Department of Commerce, the heads of other bureaus and offices in the Department of Commerce, members of Congress and staff members of House and Senate Committees, members of the NBS Advisory Panels and the NBS Statutory Visiting Committee, and representatives of the Standing Committee of the Federal Council on Science and Technology.

Speakers at the luncheon, in addition to Mr. Price, were Congressman George P. Miller (Calif.), speaking as a representative of the House Committee on Science and Astronautics, and Congressman Charles Mathias, representing the 6th District of Maryland, which includes Montgomery County, site of the Bureau's new laboratories.

Other speakers were Rear Admiral H. Arnold Karo, Director of the U.S. Coast and Geodetic Survey; Dr. Thomas Nolan, Director of the Geological Survey; Dr. Mervin J. Kelly, Chairman of the NBS Statutory Visiting Committee; Dr. Paul D. Foote, National Academy of Sciences-National Research Council coordinator of the Bureau's Advisory Committees; and Commissioner Karl E. Wallace of the U.S. Public Buildings Service.

[At the Bureau's Boulder (Colo.) Laboratories, an open house was held to commemorate the anniversary. Highlight of the occasion was a talk by Dr. J. Howard Dellinger, first chief of the Bureau's central Radio Propagation Laboratory, on "Fifty Years of Radio at the National Bureau of Standards." Dr. F. W. Brown, Director of the Boulder Laboratories, attended the Washington ceremonies.]

As part of the anniversary ceremonies, the guests visited a number of research laboratories representing a cross section of the Bureau's program. Among the research topics covered were:

NEW STANDARD OF LENGTH. The meter, now defined as 1,650,763.73 wavelengths of light from the krypton atom, is the basis for all length measurements in the United States, from atomic diameters to the breadth of

the land itself. The exhibit showed how the wavelength of light is brought into relation with the length of the international meter bar, which served as a standard from 1889 to 1960.

Mass Standards and Precision Weighing. The National standard of mass—a platinum-iridium cylinder which is a copy of the international standard kept at Sèvres, France—is the basis for all measurements of mass and force in the United States. The laboratory exhibit showed how weighing techniques are performed from micrograms to 50-lb weights. A special one-pan balance designed by the Bureau for weighing in this range was exhibited.

Standard of Frequency and Time Interval. (Boulder, Colo., Laboratories) Time and its reciprocal, frequency, can be measured with greater precision than any other physical quantity. By matching the frequency of electronic oscillators to the natural frequencies of atoms a standard of frequency has been constructed affording precise measurements to one part in a hundred billion. By counting the cycles of such oscillators a time scale can be constructed which is more than one hundred times as uniform as the earth's rotation. These highly precise standards of frequency and time-interval are made immediately available to users by radio broadcasts from stations such as WWV, WWVB, and WWVH of the Bureau. The reception of time and frequency signals from WWV was demonstrated, and a model of the cesium beam frequency standard was displayed.

Atomic Beams. NBS research on atomic beams promises to make possible a more precise wavelength standard of length. The visitors were shown apparatus that uses an atomic beam of mercury 198 to produce a spectral line nearly 10 times as sharp as the krypton 86 wavelength standard adopted in October 1960. This apparatus has been used to compare wavelengths to a precision of one part in a billion.

Gage Blocks. Mass production requires that all dimensions be related to a single standard of length. In the United States this single standard is that maintained by the Bureau. Gage blocks provide a means for industry to precisely control dimensional tolerances. The Bureau has developed procedures for calibrating master gage blocks to an accuracy of one part in 10 million. Significant progress has been made in the development both of highly stable gage blocks and of ultraprecise techniques for measuring and comparing them.

Measuring Large Forces. For calibration of force-measuring devices, the Bureau uses dead-weight machines, the largest of which has a capacity of 111,000 lb. Today new rocket engines to develop thrusts of more than a million pounds are being designed, creating an urgent demand for higher-capacity force-measurement systems in the missile and satellite programs. To meet these demands, the Bureau is designing dead-weight machines of 300,000- and 1,000,000-lb capacities to be installed at the new Gaithersburg site.

Measuring Pressures to 1,000,000 lb/in.^{2} Industry utilizes pressures as high as 1 million lb/in^2 to synthesize materials such as diamond and boron nitride. Pressures as low as 1-trillionth lb/in^2 are being measured in work on atomic accelerators, preparation of pure materials, and in constructing electronic equipment. Effective use of both high and low pressures requires a well-defined scale of pressure, standard measurement methods, and data on the properties of materials at various regions of the pressure scale. Special research programs at NBS will provide for these needs.

Plasma Thermometry. Basic research makes possible establishment of standards for measuring very high temperatures, such as occur in plasmas. Much work has been done to produce a high-temperature source that will be both stable and reproducible. The Bureau has developed a high-current density arc with a number of new features, which operates in the region from 10,000 to 20,000 °K. Temperatures in the arc are determined by measuring the intensity and shape of the spectral lines it produces.

Heat Measurements. Accurate measurement of the thermal properties of pure materials, such as thermal diffusivities, is of vital importance in the development of nuclear power and in the design of high-speed aircraft, missiles, and re-entry vehicles. The Bureau is devising techniques for accurately measuring these properties and developing reference materials to serve

Secretary of Commerce Luther H. Hodges (foreground) and NBS Director A. V. Astin examine a display of standard samples issued by the Bureau. The Bureau now distributes 60,000 samples of standard materials a year for use in controlling chemical processes and in maintaining the accuracy of apparatus and equipment.

Guests at the Bureau's 60th anniversary celebration visit the gage block calibration laboratory. Left to right: John Beers, in charge of the laboratory; Congressman John W. Davis (Georgia); John Stillman, Special Assistant to the Secretary of Commerce; Samuel Lawrence, Bureau of the Budget; C. S. McCamy, Chief of the Bureau's Photographic Research Section, acting as guide; Peter Newfeld of the gage block laboratory.

---

as standards for measurement of thermal properties in other laboratories.

THERMAL CONDUCTIVITY MEASUREMENTS AT HIGH TEMPERATURES. Rocket development and the design of nuclear power generators require accurate data on the heat conduction properties of a number of materials at high temperatures. The Bureau has developed new methods for high-temperature thermal-conductivity measurements on metals and solid and granular materials.

THE STANDARD VOLT AND FARADAY. All electrical and electronic measurements are based on the fundamental standards of resistance and electromotive force maintained by the Bureau. The NBS Standard Cell Laboratory maintains the national standard of electromotive force consisting of 44 cells of a special type. Intercomparisons of voltage units with the national standard may be made with a precision of one part in 10 million. Adjacent to the Standard Cell Laboratory visitors could see the equipment used in the redetermination of the faraday, a fundamental constant of physics and chemistry.

MEASUREMENT OF ELECTRICAL ENERGY. Public utility commissions and power companies maintain accuracy by calibrating watthour meters against master standards which are linked back to the Nation's fundamental electrical standards. Established and maintained by NBS, these national standards are known to a few parts in a million.

STANDARDS OF RADIOACTIVITY. Radiation from radioactive sources is becoming increasingly important in medicine, industry, and civil defense and atomic energy programs. All these applications require precise standards of radioactivity and calibrated radiation-measuring instruments. For measuring very low levels of radioactivity, corresponding to natural background radiation or radioactive fallout, special laboratories containing specially designed equipment were recently completed at the Bureau.

NEUTRON PHYSICS: RESEARCH AND STANDARDS. Effective use of high-energy accelerators, nuclear reactors, and controlled thermonuclear power, requires an understanding of the properties of the neutron and the way it interacts with matter. The Bureau is contributing to the advancement of knowledge in this field through the development and maintenance of standards of neutron source strength and thermal neutron flux, studies of neutron penetration and shielding, measurement of neutron dose in radiation fields, and investigations of improved neutron detectors.

RESEARCH IN LABORATORY ASTROPHYSICS. In the U.S. space program great efforts are made to fly rocket-born spectrographs to photograph the layers of the sun's atmosphere in which originate the radiations that may hold the key to long-range weather prediction. NBS laboratories make accurate measurements of atomic absorption processes that occur in these layers, thus providing the data without which the astrophysical observations cannot be interpreted quantitatively. Among the projects in laboratory astrophysics demonstrated to the visitors were: *A Wall-Stabilized Arc.* The "Fraunhofer lines" are produced in the "reversing layer" of the sun. Accurate means of measuring the transition probabilities of these lines are essential to plasma physics and astrophysics. A high-current wall-stabilized arc is used to obtain gases at temperatures from 7,000 to 15,000 °K. The emitted light is photoelectrically recorded with a spectrometer and provides information to determine temperature and particle concentrations. *The Solar Continuum.* The familiar continuous spectrum of the sun originates in the solar "photosphere," where a little known particle, the negative hydrogen ion, is responsible for producing most of the radiation. Apparatus capable of reproducing this absorption and emission of light by the hydrogen ion in the laboratory has been under use and continued refinement at NBS since 1953.

THERMODYNAMIC DATA. The compilation of data on the chemical thermodynamic properties of chemical substances is greatly needed in the development of propellants, explosives, and fuels for the defense effort; in the development of industrial processes involving chemical reactions; and in fundamental research on the physical and chemical behavior of materials. The Bureau's program in this field involves both the compilation of data from a critical review of world literature, and laboratory research to provide otherwise unavailable key information.

HIGH-TEMPERATURE PROPERTIES OF MATERIALS. One of the most active areas in the Bureau's investigations of the properties of materials is the development

of means for precisely measuring the physical and chemical properties of existing materials at very high temperatures. A current study deals with methods for determining vapor pressures and related properties of materials such as platinum and refractory oxides at temperatures above 1,800 °F.

THERMAL BEHAVIOR OF MATERIALS FOR SATELLITE SURFACES. The outer shell of a space vehicle should be so designed as to establish internal temperature conditions suitable for normal functioning of instrumentation and living occupants, if any. Because the needed data on heat radiating and absorbing characteristics of materials are not available, a number of different laboratories have set up equipment for determining these properties but their results have often been divergent. The Bureau has therefore undertaken a program for the Wright Air Development Center to develop standard techniques and instrumentation for evaluating the thermal emittance, thermal reflectance, and solar absorption of various materials.

GROWTH OF METAL CRYSTALS. NBS metallurgical research is directed toward a better understanding of the properties of existing metals to facilitate the development of improved metals and alloys to meet new requirements or to give better performance. Fundamental to an understanding of many of the properties of metals is a knowledge of the distribution and type of imperfections which may exist within their crystals. This knowledge is being sought through a study of the actual growth of metal crystals.

Remarks by Hickman Price, Jr., Assistant Secretary of Commerce, at the NBS 60th Anniversary Program, March 3

IT IS A GREAT PRIVILEGE for me to be here today on the occasion of the 60th Anniversary of the National Bureau of Standards. I take particular pleasure in extending to the Bureau the hearty congratulations of the Secretary of Commerce. Mr. Hodges also sends his best wishes to all of you here today, and he will join us later at the tea.

The Department of Commerce is charged with the responsibility of promoting the healthy growth of American business. In the world of today, the future of business is irrevocably linked with the future of science, technology, industry, and trade. Science is based upon the exchange of scientific data. Technology requires the precise measurement of physical phenomena. Our mass-production industry depends upon the interchangeability of components. Commerce and trade need an equitable basis for the exchange of goods. In all of these areas, the National Bureau of Standards makes a substantial contribution by providing the central basis for our national measurement system.

Some of you may have noticed the vault on the first floor of this building. In the vault are the kilogram and meter bar which were brought to this country from France in 1890. Then, just down the hall from this room, you may have seen models of the new Gaithersburg laboratories of the National Bureau of Standards. These two displays tell us something about the Bureau, and the vital job it must do to provide leadership in the science of measurement. In the vault, we see the historical aspects of measurement and its links with the past. In the models, we see the dynamic nature of measurement and its role in the future. In fact, the Secretary of Commerce announced this very morning that bids are now being accepted for the construction of the first two buildings of the new laboratories. The techniques, the standards, the facilities of yesteryear are not good enough to meet the demands of the space age.

The great, exciting fields of science today are nuclear technology, space exploration, miniaturization, automation, and a host of others. In every one of these areas, the Bureau is the ultimate source for uniform and accurate standards of physical measurement, and for precise data on the properties of materials. The Bureau also serves as a central agency within Government for specialized information in the fields of radio propagation, cryogenic engineering, building and fire research, and data processing systems.

The President's keen interest in scientific developments is well-known to you all, and this attitude is shared by the Secretary of Commerce. Mr. Hodges' experience as Governor of North Carolina convinced him of the interdependence of economic growth and scientific progress. Thus we take special pride in the outstanding role the Bureau of Standards has played in the advancement of science and technology in our country.

In these times, of course, no agency can afford to rest on its laurels. We have heard much lately about the "measurement pinch"—the situation in which the explosive growth of science and technology has outdistanced the measurement competence of the country. The Bureau is making every effort to alleviate this situation, and we in the Department of Commerce are determined to support the Bureau's efforts along these lines.

And so this 60th Anniversary celebration is given greater significance today for it finds the Bureau preparing to take the first major step toward construction of its new facilities, a step which should go far in helping the Bureau to meet the nation's measurement needs of tomorrow. On behalf of the Secretary of Commerce, I congratulate the National Bureau of Standards on its sixtieth birthday, as a strong and vibrant member of the Commerce family.

IMPACT PROPERTIES OF TEXTILES. Industry needs basic information on the strength and shock-absorbing properties of textiles to develop superior textiles for such uses as the cord in a jet plane tire, the fabric in body armor for protection against shell fragments, and the shroud lines and fabric of a parachute. To measure these properties, the Bureau has developed special precision equipment, utilizing high-speed photography. Effects of impact loading by projectiles travelling from 100 to over 1,000 mph are measured.

PEEK-A-BOO INFORMATION STORAGE AND RETRIEVAL. A novel perforated-card system has been developed for indexing scientific or other information by categories. Information on a specific subject is easily retrieved, either manually or by semiautomatic machine. The Bureau is using the system to increase the effectiveness of its literature searching in the field of instrumentation.

DATA PROCESSING SYSTEMS. In the field of automatic data processing systems, the Bureau serves the Government as a central research and development agency and as a readily available source of technical information. At the same time the Bureau's competence in data processing enables it more effectively to accomplish its measurement mission. SEAC, now used as a research tool for developing new computer systems and applications was the first of the high-speed computers with stored programming to go into operation in this country. When completed, PILOT will be a multipurpose computer network expected to augment the Bureau's data processing research program in such new areas as mechanized patent searching, language translation, and recognition of pictorial information.

SIMULATION OF MAN-MACHINE COMPLEXES. The particular situation being simulated is a portion of an air traffic control system which could not reasonably be brought into a laboratory. Purpose of this research is to study the interaction between the human operators and a proposed electronic environment. The techniques could be applied to studies of a wide variety of command and control systems.

RADIO PROPAGATION RESEARCH. (Boulder, Colo., Laboratories) Major segments of our defense effort, outer space program, and our civilian economy depend on the transmission of information via radio waves. The Bureau has a central responsibility within the Federal Government for collecting, analyzing, and disseminating information on the propagation of radio waves at all frequencies along the surface of the earth, through the atmosphere, and in outer space. To carry out this responsibility, the Central Radio Propagation Laboratory in Boulder, Colo., conducts research on the nature of the waves, the media through which they are transmitted, and the interaction of the waves with the media. A photographic exhibit was used to describe this program.

Note: For an earlier article on the Bureau's 60th anniversary, see NBS Marks 60th Anniversary, NBS Tech. News Bul. 45, 37 (1961).

---

[1] *A more complete description on the new Gaitherburg facilities appeared in the August 1960 issue of the Technical News Bulletin. Design of the new facilities is by the architectural firm of Voorhees Walker Smith Smith and Haines of New York City. Funds for the later construction phases have not yet been provided, but plans call for a continuing construction program leading to total relocation by the end of 1965.*

Point in Antarctic Named for Boulder Scientist

A PROMONTORY OF LAND in Antarctica has recently been named in honor of a Bureau scientist. Garth Stonehocker, a physicist at the Boulder Laboratories, has been notified that a point near Wilkes Station has been named Stonehocker Point in recognition of his contributions while a participant in the United States scientific program in Antarctica during the International Geophysical Year.

Stonehocker, who was in charge of the ionospheric program at Wilkes Station from 1957 to 1958, operated a radar-like sounder to obtain data on the ionosphere. He was one of the first to realize that solar-caused ionospheric disturbances were occasionally being detected sooner in the Antarctic than at warning stations in other parts of the world. These phenomena, designated polar cap absorption events, are now being intensively studied in both the Arctic and the Antarctic regions.

Wilkes Station was built for use during the International Geophysical Year by the United States Navy, and for the first two years (1957–59) it was staffed by United States scientists. The station is now manned by Australian personnel who have continued scientific studies of the region. Located on a rocky peninsula that juts out into the Indian Ocean, Wilkes Station was one of the most isolated of the U.S. Antarctic bases. The point named after Stonehocker is several hundred yards from the station at latitude 66°15′, longitude 110°31′ E.

Further information about this recognition came to Stonehocker in a letter from the chairman of the U.S. National Committee, International Geophysical Year 1957–58, which states: "The proposal to name this feature in your behalf was made by Dr. Carl R. Eklund, Scientific Station Leader, Wilkes Station; Special Committee on IGY Geographic Names in the Antarctic; and the U.S. Board on Geographic Names, Department of the Interior.

"Members of the U.S. National Committee for the International Geophysical Year join me in extending our congratulations for this well-earned recognition."

Publications of the National Bureau of Standards

Periodicals

Technical News Bulletin, Volume **45,** No. 4, April 1961. 15 cents. Annual subscription: $1.50, 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Basic Radio Propagation Predictions for July 1961. Three months in advance. CRPL–200, issued April 1961. 15 cents. Annual subscription $1.50, 50, 50 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75.

Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75.

Section D. Radio Propagation. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75.

Nonperiodicals

Atomic energy levels in crystals, J. L. Prather, NBS Mono. 19 (1961) 60 cents.

A spectrophotometric atlas of the spectrum of CH from 3000A to 5000A, A. M. Bass and H. P. Broida, NBS Mono. 24 (1961) 20 cents.

Development of high-temperature strain gages, J. W. Pitts and D. G. Moore, NBS Mono. 26 (1961) 20 cents.

Units of weight and measure (United States customary and metric) definitions and tables of equivalents, L. V. Judson, NBS Misc. Publ. 233 (1960) 40 cents.

Technical Notes

The following Technical Notes are available from the Office of Technical Services, U.S. Department of Commerce, Washington 25, D.C. (Order by PB number).

Quarterly radio noise data, September, October, November 1960, W. Q. Crichlow, R. T. Disney and M. A. Jenkins. NBS TN18–8 (PB151377–8) (1961) $1.75.

Prolonged space-wave fadeouts in tropospheric propagation, A. P. Barsis and M. E. Johnson. NBS TN88 (PB161589) (1961) $2.00.

Publications in Other Journals

The minima of cyclic sums, K. Goldberg, J. London Math. Soc. **35,** 262–264 (1960).

Normal approximation to the chi-square and non-central *F* probability functions, N. C. Severo and M. Zelen, Biometrika **47,** No. 3 & 4, 411–416 (1960).

Microtechnique for the infrared study of solids—diamonds and sapphires as cell materials, E. R. Lippincott, F. E. Welsh and C. E. Weir, Anal. Chem. **33,** 137–143 (Jan. 1961).

Definitions relating to metals and metalworking, M. R. Meyerson and S. J. Rosenberg, Am. Soc. Metals Handb. **1,** 1–41, (1961).

Stress-ruptured tests at 1350° F on type 304 stainless steel, W. D. Jenkins, W. A. Willard, and W. J. Youden, ASTM Bul. **1,** No. 2, 104–108 (Feb. 1961).

Techniques in calorimetry. I. A noble-metal thermocouple for differential use, E. D. West, Rev. Sci. Inst. **31,** No. 8, 896–897 (Aug. 1960).

Paratellurite, a new mineral from Mexico, G. Switzer and H. E. Swanson, Am. Mineralogist **45,** 1272 (Nov.-Dec. 1960).

Evaluation of micrometer and miscrosopical methods for measuring thickness of floor coverings, E. Horowitz, J. Mandel, R. J. Capott, and T. H. Boone, ASTM Bul. **1,** No. 2, 99–102 (Feb. 1961).

Electrodeposition of metals from nonaqueous media, A. Brenner, Chapter on Electrodeposition, Encyclopedia Chem. Tech. 2d Suppl., 315–324 (Jan. 1961).

The nature, cause and effect of porosity of electrodeposits **VI.** Note on a magnetic method of detecting corrosion currents, F. Ogburn and W. H. Roberts, Plating **48,** No. 2, 168–169 (Feb. 1961).

The height of maximum luminosity in an auroral arc, F. E. Roach, J. G. Moore, E. C. Bruner, Jr., H. Cronin, and S. M. Silverman, J. Geophys. Research **64,** No. 11, 3575–3580 (Nov. 1960).

Physical metallurgy and mechanical properties of materials: Ductility and the strength of metallic structures, J. M. Frankland, J. Eng. Mech. Div. Proc. Am. Soc. Civil Engr., **86,** No. EM 6, 45–52 (Dec. 1960).

Cryogenic impurity adsorption from hydrogen, M. J. Hiza, Chem. Engr. Progress **55,** No. 10, 68–71 (Oct. 1960).

Nomenclature for standards of radioactivity, A. G. McNish, Intern. J. Appl. Radiation and Isotopes **8,** 145–146 (Jan. 1960).

Lithium, M. R. Meyerson, Am. Soc. for Metals Handb. **1,** 1213 (1961).

Tensile cryostat for the temperature range 4° to 300° Kelvin, R. M. McClintock and K. A. Warren, ASTM Bul. **1,** No. 2, 95–98 (Feb. 1961).

Standards and testing, the key to quality, B. E. Foster, Eng. Bul., Purdue Univ. **XLIV,** No. 6, 54–62 (Nov. 1960).

A radio-frequency permittimeter, R. C. Powell and A. L. Rasmussen, IRE Trans. Instrumentation **I–9,** No. 2, 179–184 (Sept. 1960).

Subgroups of the modular group and sums of squares, M. Newman, Am. J. Math. **82,** No. 4, 761–778 (Oct. 1960).

Phase equilibria in the system cadmium oxide-niobium oxide, R. S. Roth, J. Am. Ceram, Soc. **44,** No. 1, 49–50 (Jan. 1961).

Redetermination of the chromium and nickel solvuses in the chromium-nickel system, C. J. Bechtoldt and H. C. Vacher, Trans. Metallurgical Soc., Am. Inst. Mining Engrs. **221,** 14–18 (Feb. 1961).

A modulated subcarrier technique of measuring microwave phase shifts, G. E. Schafer, IRE Trans. Instrumentation **I–9,** No. 2, 217–219 (Sept. 1960).

An analysis of the accumulated error in a hierarchy of calibrations, E. L. Crow, IRE Trans. Instrumentation **I–9,** No. 2, 105–114 (Sept. 1960).

Hydrogen-bonding in calcium-deficient hydroxyapatites, A. S. Posner, J. M. Stutman, and E. R. Lippincott, Nature **188,** No. 4749, 486–487 (Nov. 1960).

Hydrogen sulfide precipitation of the elements from 0.2–0.5 normal hydrochloric acid, J. I. Hoffman, Chemist Analyst **50,** No. 1, 30 (March 1961).

Dielectric constant and dielectric loss of TiO_2 (Rutile) at low frequencies, R. A. Parker and J. H. Wasilik, Phys. Rev. **120,** No. 5, 1631–1637 (Dec. 1960).

Generating functions for formal power series in non-commuting variables, K. Goldberg, Proc. Am. Math. Soc. **11,** No. 6, 988–991 (Dec. 1960).

Tests for regression coefficients when errors are correlated, M. M. Siddiqui, Ann. Math. Stat. **31,** No. 4, 929–938 (Dec. 1960).

Application of the method of polarized orbitals to the scattering of electrons from hydrogen, A. Temkin and J. C. Lamkin, Phys. Rev. **121,** No. 3, 788–794 (Feb. 1961).

A recommended standard resistor-noise test system, G. T. Conrad, Jr., N. Newman and A. P. Stansbury, IRE Trans. Component Parts, **CP–7,** No. 3, 71–88 (Sept. 1960).

On the absorption spectrum of CF_2 and its vibrational analysis, D. E. Mann and B. A. Thrush, J. Chem. Phys. **33,** No. 6, 1732–1734 (Dec. 1960).

A transfer instrument for the intercomparison of microwave power meters, G. F. Engen, IRE Trans. Instrumentation **I–9,** No. 2, 202–208 (Sept. 1960).

Standards and measurements of microwave surface impedance, skin depth, conductivity and *Q,* H. E. Bussey, IRE Trans. Instrumentation **I–9,** No. 2, 171–175 (Sept. 1960).

UNITED STATES
GOVERNMENT PRINTING OFFICE
DIVISION OF PUBLIC DOCUMENTS
WASHINGTON 25, D.C.

OFFICIAL BUSINESS

PENALTY FOR PRIVATE USE TO AVOID
PAYMENT OF POSTAGE, $300
(GPO)

Publications (*Continued*)

Plating gun bores, V. A. Lamb and J. P. Young, Ordnance **XLV**, No. 245, 725–727 (March-April 1961).

Integrated starlight over the sky, F. E. Roach and L. R. Megill, Astrophys. J. **133**, No. 1, 228–242 (Jan. 1961).

Low temperature static seals using elastomers and plastics, D. H. Weitzel, R. F. Robbins, G. R. Bopp, and W. R. Bjorklund, Rev. Sci. Inst. **31**, No. 12, 1350–1351 (Dec. 1960).

FM and SSB radiotelephone tests on a VHF ionospheric scatter link during multipath conditions, J. W. Koch, W. B. Harding and R. J. Jansen, IRE Trans. Commun. Systems **CS–8**, No. 3, 183–186 (Sept. 1960).

Propagation of error in a chain of standards, A. G. McNish and J. M. Cameron, IRE Trans. Instrumentation **I–9**, No. 2, 101–104 (Sept. 1960).

Absolute measurement of temperatures of microwave noise sources, A. J. Estin, C. L. Trembath, J. S. Wells, and W. C. Daywitt, IRE Trans. Instrumentation **I–9**, No. 2, 209–213 (Sept. 1960).

Electron scattering in high magnetic field, A. H. Kahn, Phys. Rev. **119**, No. 4, 1189–1192 (Aug. 1960).

Measurement of reflections and losses of waveguide joints and connectors using microwave reflectometer techniques, R. W. Beatty, G. F. Engen and W. J. Anson, IRE Trans. Instrumentation **I–9**, No. 2, 219–226 (Sept. 1960).

Microwave spectrum of cis-difluoroethylene, V. W. Laurie, J. Chem. Phys. **34**, No. 1 (Jan. 1961).

Surface roughness of gold castings, J. J. Barone, R. L. Huff and G. Dickson, Dental Progr. **1**, No. 2, 78–84 (Jan. 1961).

The sensitivity of photographic film to 3-Mev neutrons and to thermal neutrons, M. Ehrlich, Health Phys. **4**, 113–128 (1960).

A prototype rubidium vapor frequency standard, R. J. Carpenter, E. C. Beaty, P. L. Bender, S. Saito, and R. O. Stone, IRE Trans. Instrumentation **I–9**, No. 2 (Sept. 1960).

Low even configurations in the first spectrum of thorium (Th I), R. E. Trees, Physica **26**, 353–360 (1960).

Comparison measurements with intensity standards for high energy bremsstrahlung, J. S. Pruitt and W. Pohlit, Z. Naturforschung **15b**, No. 9, 617–619 (1960).

High-disperson spectra of Jupiter, C. C. Kiess, C. H. Corliss and H. K. Kiess, Astrophys. J. **132**, No. 1, 221–231 (July 1960).

Propagation of electromagnetic waves along a thin plasma sheet, J. R. Wait, Can. J. Phys. **38**, 1586–1594 (1960).

Preparation and properties of aromatic fluorocarbons, W. J. Pummer and L. A. Wall, J. Chem. Engrs. Data **6**, No. 1, 76–78 (Jan. 1961).

Stability of thermoset plastics at high temperatures, S. L. Madorsky and S. Straus, Modern Plastics **38**, 134–140 (Feb. 1961).

Immiscibility and the system lanthanum oxide-boricoxide, E. M. Levin, C. R. Robbins and J. L. Waring, J. Am. Ceram. Soc. **44**, No. 2, 87–91 (Feb. 1961).

Publications for which a price is indicated are available only from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. (foreign postage, one-fourth additional). Reprints from outside journals and the NBS Journal of Research may often be obtained directly from the authors.

NBS to Conduct Course in Radio Propagation

THE BUREAU will present a three-week course in Radio Propagation this summer. The problems of sending a radiowave through the lower and upper atmosphere will be considered in two separate sections, which may be taken individually or in succession. The first, a one-week course in Tropospheric Propagation, will be offered July 31 to August 4; The second, a two-week course in Ionospheric Propagation, will be offered August 7 to 18. The problem of "static" or radio noise—of atmospheric, man-made, or cosmic sources—will be considered in the section on Ionospheric Propagation. In both sections the continuing emphasis will be on those elements of propagation which affect system design and frequency allocation.

The course is designed to give scientists and engineers from universities, industry, and government agencies access to the latest advances in radio propagation research and show how this knowledge can best be applied to the design of systems for radio communication and navigation. It will consider the entire range of useable radio frequencies and extend into the types of propagation which are being explored for the future.

Prerequisites for the course are a bachelor's degree in electrical engineering, physics, or other suitable academic or practical experience. The tuition will be $100 for Tropospheric Propagation, $200 for Ionospheric Propagation, or $300 for the entire course.

Registration will be limited and early application should be made to ensure consideration. Formal registration will be held in the lobby of the NBS Radio Building beginning at 7:00 a.m. on the first day of each section (July 31 for Tropospheric Propagation, August 7 for Ionospheric Propagation). Further details of the course and registration forms are available from: Edmund H. Brown, Educational Director, Boulder Laboratories, National Bureau of Standards, Boulder, Colo.

U.S. GOVERNMENT PRINTING OFFICE: 1961 O—591904